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MANUFACTURING METHODS AND TECHNOLOGY PROGRAM ZINC SELENIDE BLAN--ETC(U)

FEB 79 R N DONADIO, J F CONNOLLY DAAB07-78-C-2038

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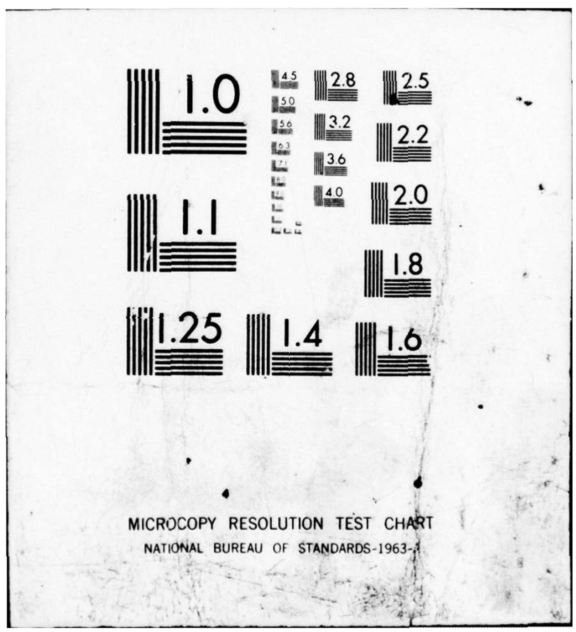
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Second Quarterly Progress Report

MANUFACTURING METHODS & TECHNOLOGY PROGRAM
ZINC SELENIDE BLANKS FOR WINDOWS AND LENS ELEMENTS

1 November 1978 to 31 January 1979

Placed by
US Army Electronics Research & Development Command
Night Vision and Electro-Optics Laboratory
Fort Belvoir, VA 22060

Contract No. DAAB07-78-C-2038

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Contractor
Raytheon Company
Research Division
Waltham, Massachusetts 02154

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Two full-scale zinc selenide deposits were successfully conducted using the evaporator system. The optical and mechanical quality of the material produced appears to be the equivalent of that made in the reservoir system.

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MANUFACTURING METHODS & TECHNOLOGY PROGRAM
ZINC SELENIDE BLANKS FOR WINDOWS AND LENS ELEMENTS

Second Quarterly Progress Report
1 November 1979 to 31 January 1979

Object of Study

The objective of this manufacturing methods and technology program is to establish the capability to manufacture high volume zinc selenide blanks for infrared windows and lens elements.

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ABSTRACT

Two full-scale zinc selenide deposits were successfully conducted using the evaporator system. The optical and mechanical quality of the material produced appears to be the equivalent of that made in the reservoir system.

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GLOSSARY

Absorption Coefficient - Fraction of energy lost while traversing a path-length of one centimeter through a material.

Chemical Vapor Deposition - A process by which chemicals are reacted in the vapor phase to form a compound.

Deposition Temperature - Temperature of the reaction zone in which the chemical vapor deposition takes place.

Evaporator - Apparatus used to form a vapor (or gas) from a solid (or liquid).

Flexural Strength - Maximum fiber stress a material will withstand before rupture in bending.

Image Spoiling Characteristics - That property of a transparent material that defines the ability to resolve discrete images.

Retort - High temperature container used to hold liquid zinc.

Substrate - A form on which material is deposited, sometimes called a mandrel.

Zinc Reservoir System - Apparatus containing one or several liquid zinc retorts and associated monitoring and controlling devices.

1.0 PURPOSE

The purpose of this manufacturing and methods technology program is to establish an automated production process for the fabrication of high optical quality zinc selenide.

The program is of seventeen months duration and is sponsored by the United States Army Electronics Research and Development Command. It addresses itself to the further automation of an existing production process for the chemical vapor deposition of zinc selenide. Raytheon Company has successfully developed the techniques and facilities to fabricate state-of-the-art CVD zinc selenide in large sizes. It is anticipated that with improvements in automated processing the price for standard size lens blanks will be reduced to 50 percent of the catalog price. In addition, the use of a curved substrate may further reduce the price of the color correcting lens shown in Figure 1 to less than $200 each for large volume purchases.

The program has been divided into three phases. In the first phase, zinc selenide test blanks will be produced using the existing process. In Phase II of the program the zinc reservoir system will be replaced with an automated external zinc supply, and blanks will be deposited for confirmation of the optical and mechanical characteristics of the material. The third phase of the program will demonstrate the production capability of a pilot line to manufacture high-quality zinc selenide blanks at four-hundred and eight-one (481) units per month.

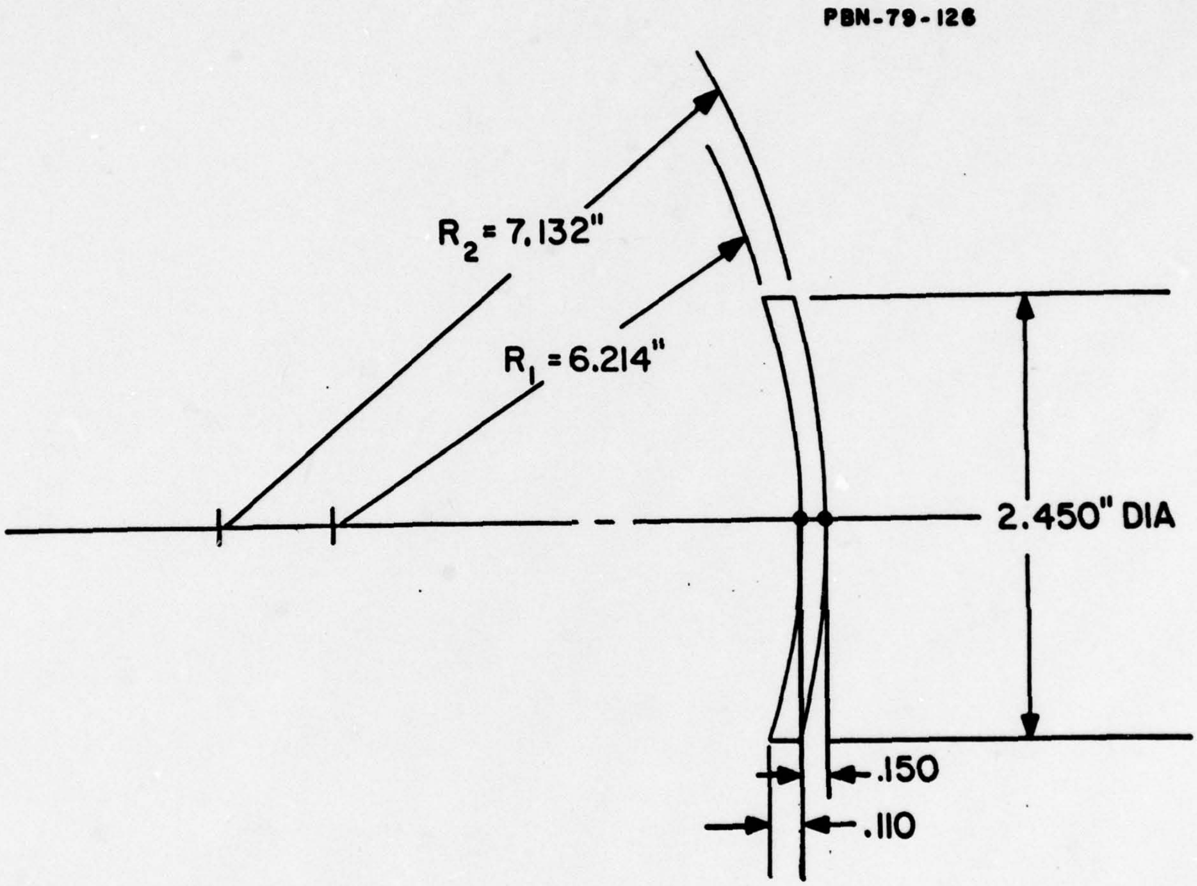

Figure 1. Zinc Selenide Compensating Lens.

2.0 NARRATIVE AND DATA

2.1 Pre-Engineering Deposit

An 84 hour zinc selenide deposit was conducted prior to the engineering sample deposit to further evaluate the performance of the deposition system. It was also felt that a moderately long run should be made to yield zinc selenide with sufficient thickness to measure the optical and mechanical properties of material made by this new method. The deposit was made in a 17 by 9 by 60 inch graphite box mandrel. The deposition conditions for this pre-engineering deposit are presented in Table 1.

The resultant deposit showed adequate overall thickness and thickness distribution. Figure 2 displays a thickness profile for Plate A, one of the two 17 by 60 inch plates deposited. Test samples were taken from the top, middle, and bottom of the deposited plates and tested for infrared transmittance. Figures 3 thru 5 present the infrared transmittance curves for these three locations. The average absorption coefficient (including surface absorption) was measured at 0.001 cm^{-1}.

Flexural test specimens were prepared from the top and bottom sections of the deposit. The zinc selenide was fabricated into 1/8 by 1/4 by 2-½ inch beams and tested in flexure, using 4-point loading over a 2 in. span. The results presented in Table 2 show slightly higher strength values than typically observed. The 730^{o}C deposition temperature for this deposit is the main factor for the increase in strength. A finer grained structure is produced at this lower deposition temperature, yielding a higher fracture strength. (Standard deposition temperature for zinc selenide is 750^{o}C; producing $\sim$ 7500 psi fracture strength with optimum optical properties.)

Examination of the evaporation system after the deposit showed no evidence of leakage to the surrounding furnace, nor zinc condensate in the evaporator chain. No malfunctions of any kind were detected throughout the deposit.

TABLE 1

DEPOSITION CONDITIONS

Run No.	Temp (^{o}C)	Furnace Pressure (torr)	H_2Se Usage (lpm)	Zinc Usage (lpm)	Deposition Time (hrs)
Pre-enginrg	730	24	4.0	700	84
Engrg. deposit	730	24	4.2	735	327

(inch)	B	C	D	E	F	G
60						
56		.122	.130	.129	.120	.083
52		.134	.143	.141	.130	.074
48	.098	.150	.151	.152	.140	.086
44	.104	.168	.173	.166	.150	.097
40	.132	.189	.196	.180	.165	.111
36	.154	.216	.207	.195	.184	.136
32	.193	.240	.233	.215	.217	.151
28	.238	.271	.244	.237	.253	.214
24	.298	.299	.258	.247	.280	.260
20	.328	.329	.296	.288	.313	.321
16	.330	.346	.326	.318	.323	.324
12	.324	.347	.336	.333	.344	.314
8	.237	.299	.307	.294	.294	.238
4	.279	.346	.360	.314	.317	.219
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Figure 2. Thickness profile, Plate A Pre-Engineering Deposit.

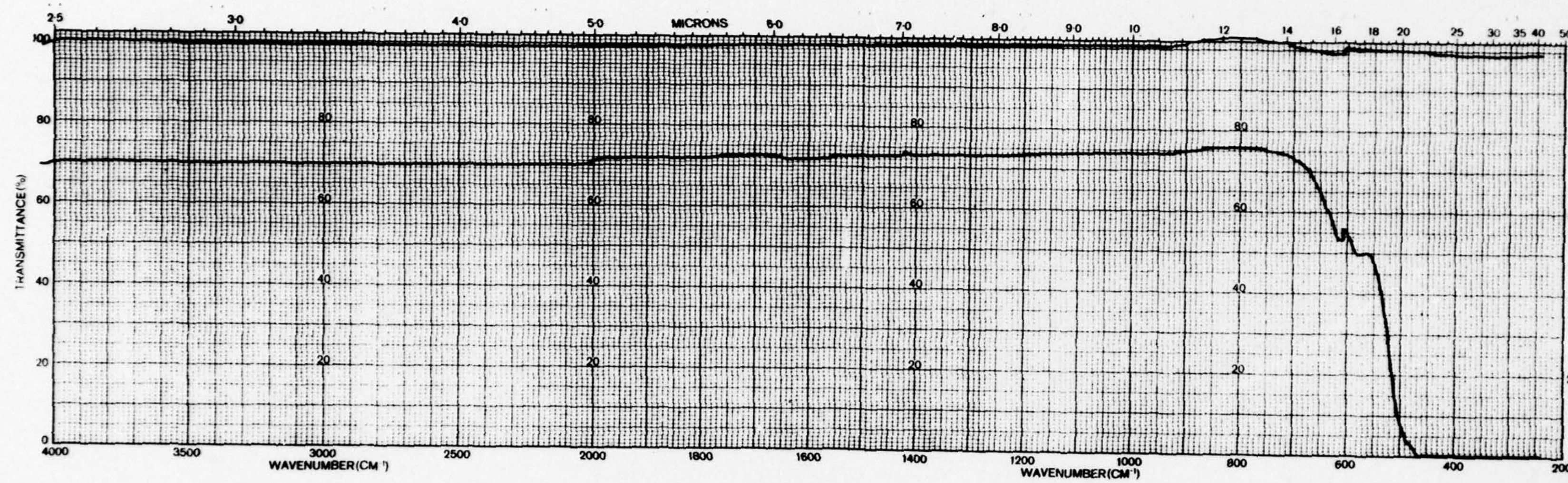

Figure 3. Infrared Transmittance, Plate C, Pos. 2M, t = 0.261 in.

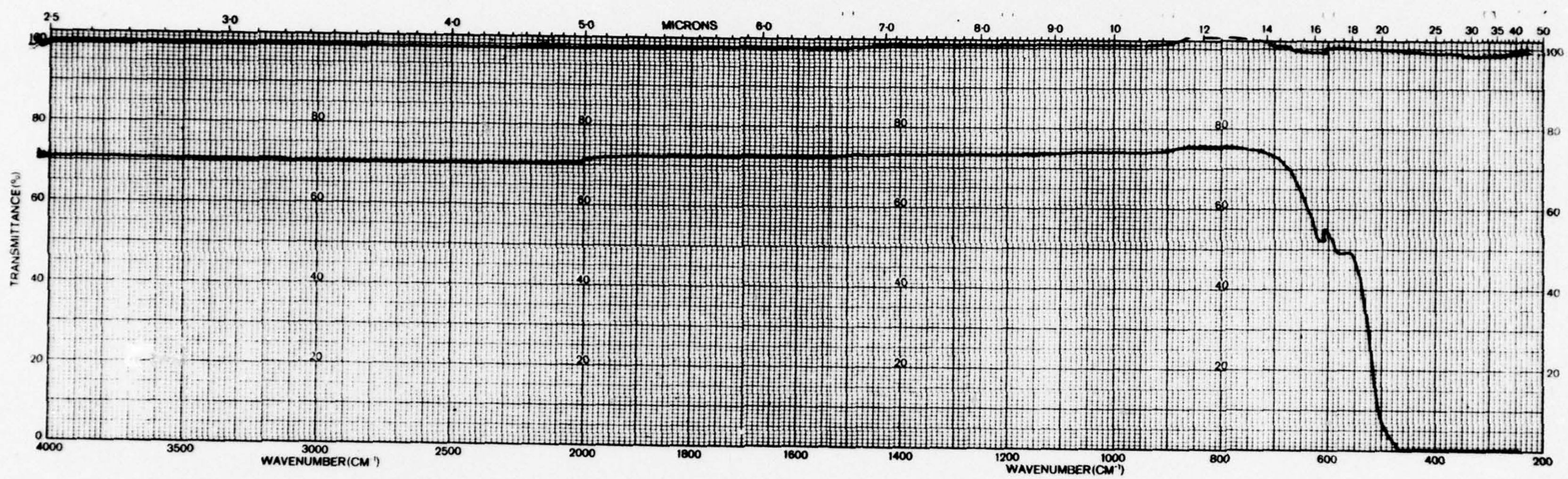

Figure 4. Infrared Transmittance, Plate D, Pos. 28M, t = 0.278 in.

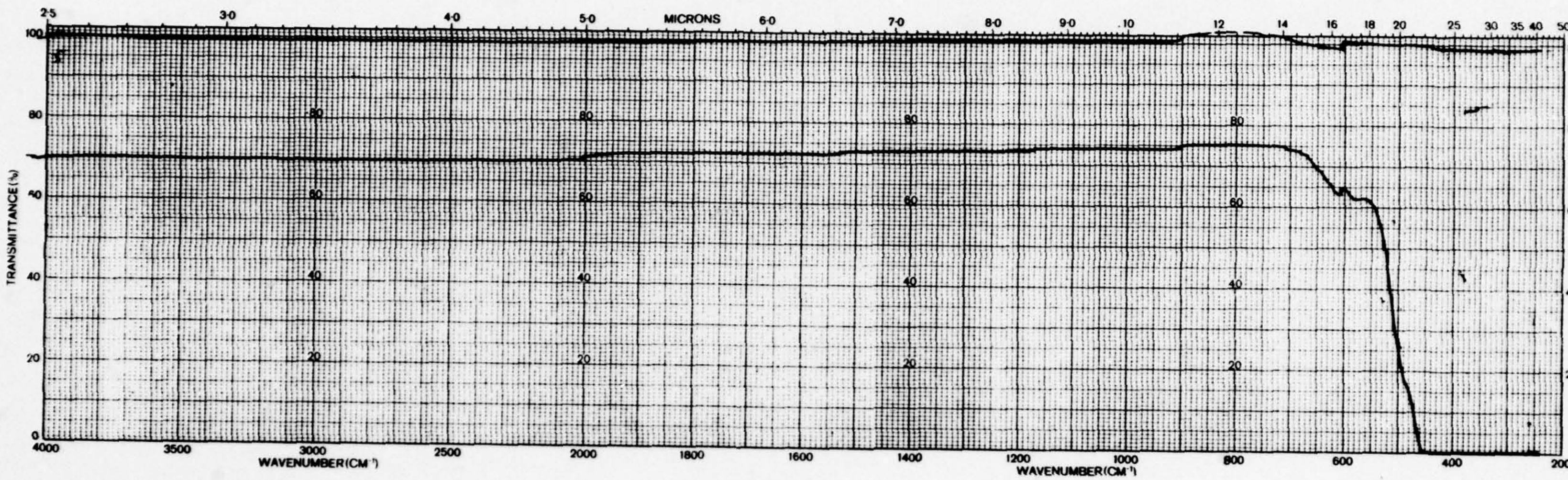

Figure 5. Infrared Transmittance, Plate D, Pos. 52M, t = 0.120 in.

Table 2

FLEXURAL STRENGTH

(4-point loading)

TOP

Specimen No.	Strength ($X\ 10^3$ psi)
1	8.5
2	8.8
3	11.9
4	9.6
5	10.0
6	7.4
7	11.5
8	10.9
9	7.3
10	8.8
Avg	9.5 ± 1.6

BOTTOM

Specimen No.	Strength ($X\ 10^3$ psi)
1	8.8
2	9.3
3	10.2
4	10.0
5	8.7
6	7.7
7	10.0
8	8.5
9	12.2
10	11.1
Avg	9.7 ± 1.3

2.2 Engineering Sample Deposit

2.2.1 Sample requirements

This program requires that 12 chemical vapor deposited zinc selenide blanks be fabricated and tested. The blanks shall be fabricated to 75 mm diameter by 9.7 mm thick. Dimensional tolerances on all samples shall be $^{+2}_{-0}$ mm. The blanks will be tested to meet the following specifications:

a) Transmission. The uncoated transmission of the blanks shall be greater than 58% over the wavelength region 8 to 13 micrometers at normal incidence. Over the wavelength region 0.6 to 1.1 micrometers the transmittance shall be greater than 43%.

b) Absorption The absorption over the 8-12 micrometer region shall be less than 0.01 per centimeter at 10.6 um.

c) Parallelism. The provided blank shall have maximum allowable wedge of 10 minutes.

d) Strain. The distribution of permanent strain shall be symmetrical and the birefringence resulting from permanent strain shall not produce more than 10 nanometers relative retardation or path difference per centimeter of a transmitted narrow band light source.

e) Chips and fractures. A vented fracture exceeding 10 mm in length or aiming at the center of the blank will be rejected. Blanks having pressure or fire cracks deeper than 1 mm will be rejected. Other surface irregularities, pits, or cracks will not extend into an envelope defined by the minimum allowable dimensions.

f) Scatter. The angular spread of a focused spot on a blank will not increase by more than 15 percent over the angular spread of the same spot without the sample in the beam over the wavelength region 0.6 to 1.2 micrometers. The angular spread over the wavelength region 8 to 12 micrometers will be less than 2 percent.

g) Identification and marking. Each blank will be individually bagged and the bag marked in accordance with MIL-STD-130 with the type of material, size, individual run number identification, and manufacturer's name or code symbol.

2.2.2 Deposition

A 327-hour zinc selenide deposit was conducted to yield the required engineering samples. The deposit was made using the 17 by 9 by 60 inch mandrel, similar to the Pre-Engineering deposit. Deposition conditions for this deposit are presented in Table 1.

Figure 6 displays the thickness profile of one of the plates from the Engineering deposit. Samples were located from the top of plate C and fabricated into the specified blank dimensions. The optical quality of the zinc selenide was good, however, a portion of the latter deposited material contained a visibly scattering layer. The layer did not interfere with locating the engineering samples and was outside the required thickness to produce a zinc selenide blank.

2.2.3 Tests on engineering samples

All twelve (12) engineering samples were tested and found to meet: dimensional requirements, specified chip and fracture criteria, and spectral transmittance requirements in the infrared and the visible. Typical transmittance curves are presented in Figures 7 and 8 for the infrared and visible spectra, respectively.

Engineering samples No. ENG-11 and ENG-12 were tested for absorption coefficient at 10.6 um. Each sample was measured in three locations and the average absorption coefficient (including surface absorption) was determined. Absorption coefficients of $1.98 \times 10^{-3} cm^{-1}$ and $1.68 \times 10^{-3} cm^{-1}$ were measured for samples No. ENG-11 and ENG-12, respectively. The thickness of these two samples was 1.08 cm.

(in.)	B	C	D	E	F	G
60						
56	.379	.483	.526	.565	.510	.353
52	.366	.524	.583	.590	.573	.352
48	.369	.558	.596	.645	.650	.390
44	.367	.558	.637	.683	.688	.423
40	.375	.593	.676	.749	.759	.508
36	.360	.636	.754	.801	.814	.554
32	.426	.715	.992	.858	.913	.713
28	.568	.882	.875	.919	.999	.863
24	.845	1.08	1.0+	.980	1.0+	.915
20	1.1	1.2+	1.0+	1.0+	1.1+	1.1+
16	.994	1.3+	1.1+	1.1+	1.2!	1.1+
12	1.3+	1.4+	1.2+	1.1+	1.2+	1.0+
8	.919	1.3+	1.1+	1.1+	1.1+	.855
4	.777	1.6+	1.0+	1.0+	1.6+	.740
0						

flow ↑

1" | 3"

Figure 6. Thickness Profile, Plate A, Engineering Deposit

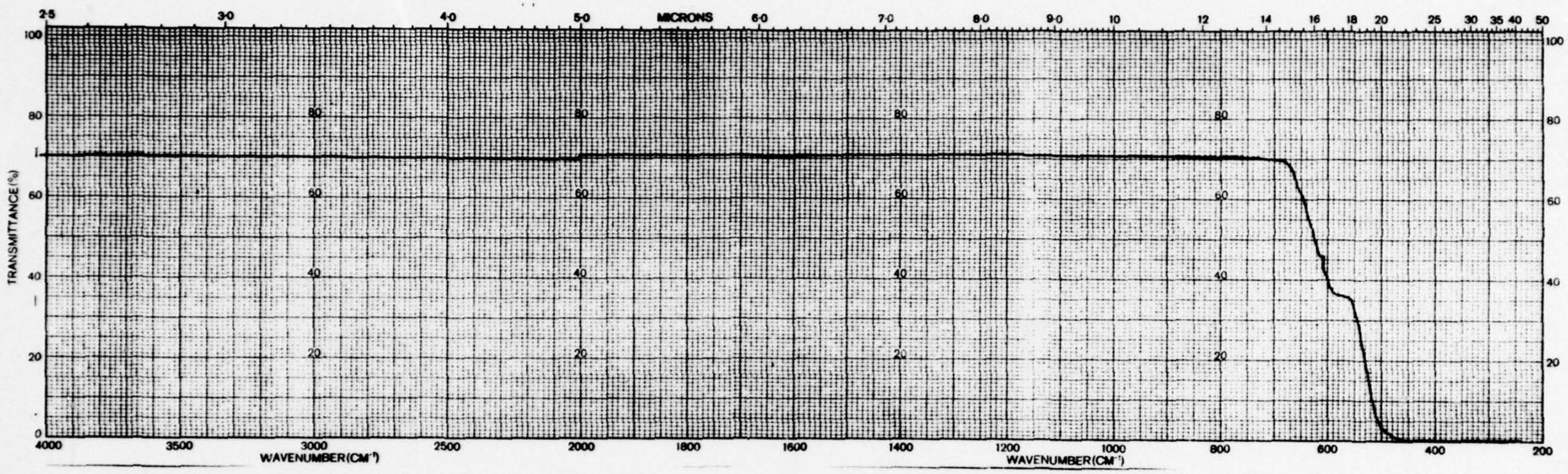

Figure 7. Infrared Transmittance of Sample ENG-1.

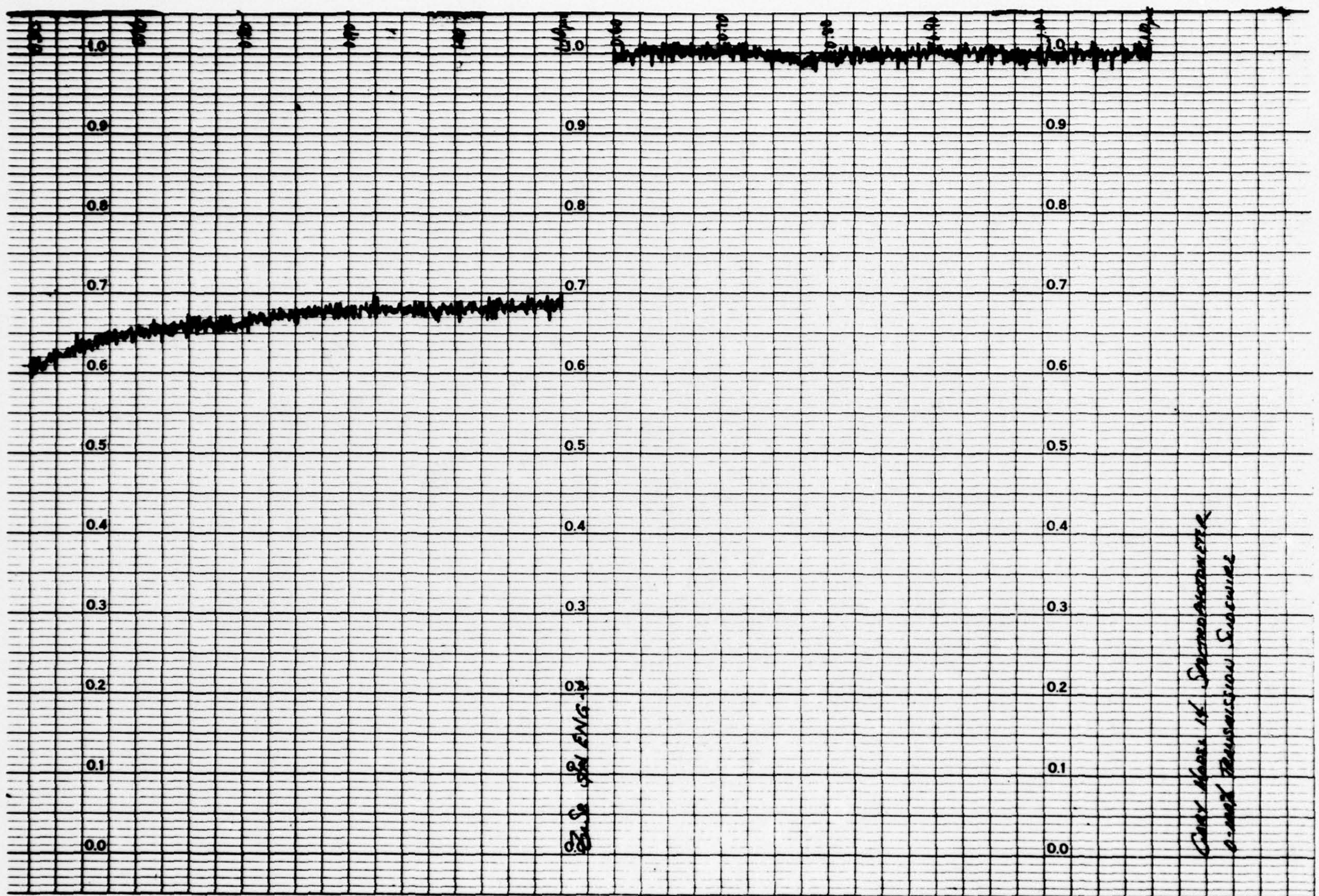

Figure 8. Transmittance From 0.6 to 1.1 μm of Sample ENG-1.

All twelve zinc selenide blanks tested for strain showed no relative retardation. Localized birefringence is present in individual crystallites, however, no larger order birefringence was discernible.

Table 3 presents the image spoiling data from samples No. ENG-11 and ENG-12. These results are discussed in Section 3.0 - MEETINGS.

2.3 Substrate Modifications

An alternative method of depositing zinc selenide on a curved substrate rather than on a flat has been recently investigated under this program. In this method, the zinc selenide is deposited to near net shape on one side. Figure 9 displays the concept of the curved mandrel. This method of producing lenses will shorten the length of the required deposit, resulting in a direct cost reduction, and will reduce the fabrication cost of the finished lens. This deposition technique is being implemented in the confirmatory and pilot runs. New curved substrates have been fabricated for use in ths confirmatory deposit.

3.0 MEETINGS

A meeting was held on 26 January 1979 at the Research Division with Raytheon personnel involved in the program and Mr. D. Helm and Mr. A. Efkeman, both of Fort Belvoir. The engineering sample test results and program requirements were discussed at this time. One area of concern was the failure of sample No. ENG-12 to meet the required image spoiling criteria (allowable image growth of 2%; 3.1% tested for sample ENG-12). The interferograms for this sample were examined, revealing a surface figure that does not meet the polishing specifications required for testing. It appears that this is the cause for the excessive image broading seen in this sample. Mr. Efkeman has consented to further test this sample on his image spoiling equipment to determine if the problem is in the material quality or sample preparation.

TABLE 3

IMAGE SPOILING DATA

Sample No.	Image Width (µrad) No. Sample	Image Width (µrad) With Sample	% Image Growth	Spectra
ENG-11	155.7	158.6	1.8	8-12 µm
ENG-12	155.7	160.5	3.1	
ENG-11	16.8	17.9	6.5	0.6-1.1 µm
ENG-12	16.8	17.9	6.5	

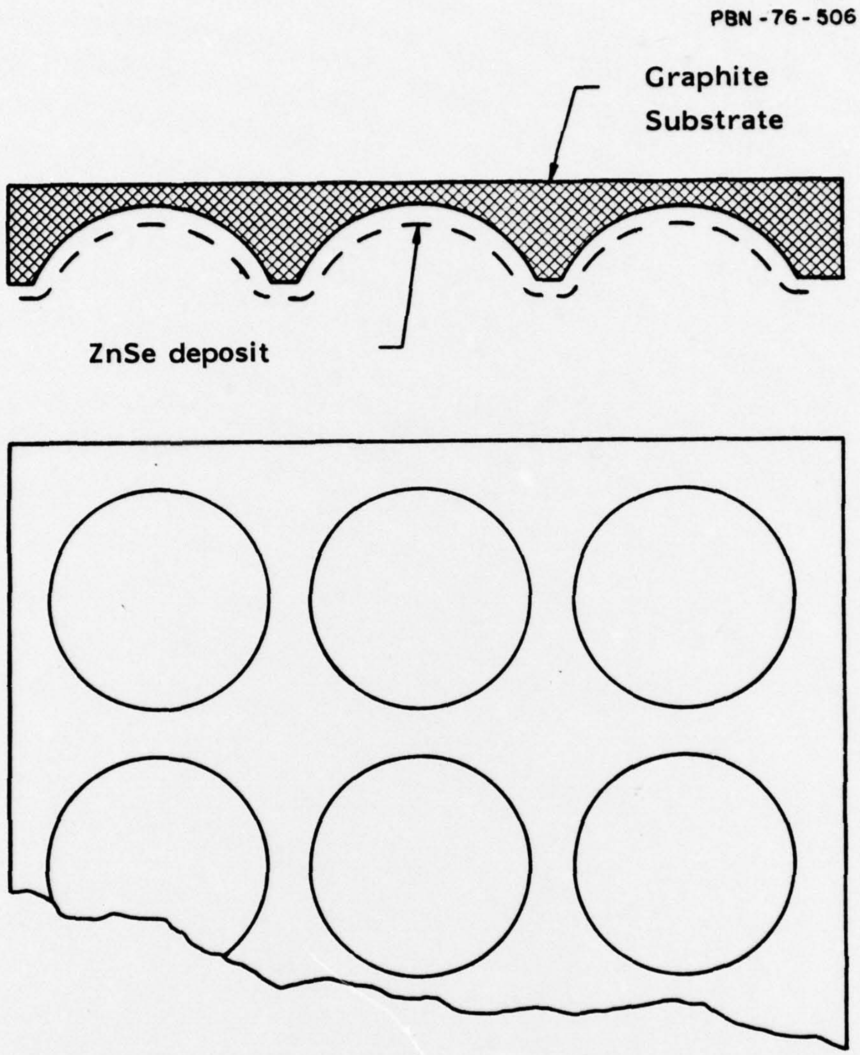

Figure 9.   Curved Mandrel.

The twelve (12) engineering samples and the Engineering Test Report were submitted at this meeting.

4.0 CONCLUSIONS

The evaporator system has performed successfully in two full-scale deposits producing zinc selenide of the same quality of that made with the reservoir system.

5.0 PROGRAM FOR THE NEXT INTERVAL

The confirmatory deposit will be set up and deposited during the next reporting period. The zinc selenide from this deposit will be cut and fabricated into the required test blanks. Testing and property evaluation on these samples will be conducted.

6.0 PUBLICATIONS

There were no publications during this reporting period.

7.0 PERSONNEL

The following is the worked manhours for key personnel on this program:

Name	Mahours During Report Period
Mr. R. Donadio	21.0
Mr. T. Varitimos	24.0
Research Technicians	268.0
Publications	18.5
Total	331.5

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